

Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) **EP 0 916 709 A2**

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:
19.05.1999 Bulletin 1999/20

(51) Int Cl.⁶: **C09D 163/00, C09D 5/03,
C08G 59/18**

(21) Application number: **98308757.8**

(22) Date of filing: **27.10.1998**

(84) Designated Contracting States:
**AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE**
Designated Extension States:
AL LT LV MK RO SI

(30) Priority: **04.11.1997 US 964242
07.07.1998 US 111419**

(71) Applicant: **MORTON INTERNATIONAL, INC.
Chicago, Illinois 60606 (US)**

(72) Inventors:
• **Muthiah, Jeno
Wernersville, Pennsylvania 19565 (JP)**

• **Teti, Jeremiah J.
Reading, Pennsylvania 19606 (JP)**
• **Schlessman, Jacquelyn M.
Temple, Pennsylvania 19560 (JP)**

(74) Representative:
**Bankes, Stephen Charles Digby et al
BARON & WARREN
18 South End
Kensington
London W8 5BU (GB)**

(54) **A one-component, low temperature curable coating powder**

(57) A mixture of a self-curing epoxy resin having an equivalent weight of from 100 to 700, a melt viscosity of from 200 to 2000 centipoise at 150°C and a low temperature curing agent is extruded as one component at a

temperature below 105°C and the extrudate is cooled and pulverized to form a low temperature curable coating powder. The powder cures at a temperature of from 107 to 149°C and produces a coating having an exceptionally smooth surface with either a low or high gloss.

EP 0 916 709 A2

Description**BACKGROUND OF THE INVENTION**

5 [0001] This invention relates to a powder coating system in which a mixture of an epoxy resin and a low temperature curing agent is extruded as one component and pulverized to form a low temperature curable coating powder. The powder cures at a temperature of from about 225 to about 300°F and produces a coating having an exceptionally smooth surface with either a low or high gloss. This invention also relates to the electrostatic coating of massive metal articles and wood and low temperature curing to achieve a pleasing smoothness. It also relates to a method for com-

10 [0002] Traditionally, coating powders have been made by the extrusion of a mixture of resins and curing agents to obtain a homogeneous mixture and then grinding the extrudate and screening the comminuted product to obtain the desired particle sizes and particle size distribution. The powder is then electrostatically sprayed onto a substrate, 15 traditionally a metal substrate, and cured at temperatures much higher than 200°F. Achieving a powder coating composition that will cure on heat sensitive substrates at less than 300°F in less than 5 minutes has long been a goal of the industry. The curing of powder coatings on materials such as wood, plastic, and the like has been limited by the fact that the extrusion of a mixture of a resin and a low temperature curing agent, i.e., one that is active at 250°F or less, would cause the coating powder to gel in the extruder because the extrusion typically generates enough heat to 20 raise the temperature to 200°F or higher.

[0003] This problem has been avoided by extruding the resin and a small amount of catalyst or low temperature curing agent, grinding the extrudate, and then blending the resultant powder with an additional amount of the curing agent in powder form, according to the disclosure in co-pending, commonly assigned application Serial No. 810,745, filed March 4, 1997.

25 [0004] The high temperatures that are generated by the extrusion of a curing agent with a resin are caused in part by the fact that the temperature must be sufficient to facilitate the mixing of resins having initially high melt viscosities. Even higher temperatures are produced by the friction arising from the mixing of the still highly viscous molten resins with curing agents that are solid at room temperature.

30 [0005] Douglas S. Richart said in his article published in the April, 1996 issue of POWDER COATINGS, that the coating of wood with a low temperature cure powder is next to impossible because the coating must be cured at a temperature below 200°F and the resin must have a flow temperature of about 10 to 20 degrees lower than that. Resins having lower melt viscosities, however, generally have lower glass transition temperatures. The presence of a resin having a very low T_g in the powdered extrudate increases the tendency to sinter and thus decreases the blocking resistance of the powder during storage at the normal maximum temperature of about 80°F (27°C).

SUMMARY OF THE INVENTION

[0006] It is an object of this invention, therefore, to provide a method for extruding a one-component low temperature curable coating powder.

40 [0007] It is a related object of this invention to provide a one-component low temperature curable coating powder that has satisfactory blocking resistance during storage at normal temperatures.

[0008] It is a related object of this invention to provide a one-component low temperature curable coating powder for heat sensitive substrates and massive metal articles.

45 [0009] It is another object of this invention to provide a low temperature process for producing a smooth, high gloss coating on wood.

[0010] These and other objects of the invention which will become apparent from the following description are achieved by a method for preparing a thermosetting coating powder comprising extruding a mixture of an epoxy resin having a melt viscosity of from about 200 to about 2000 centipoise at 150°C and a curing agent which is solid at 80°F and latent at an extrusion temperature of from about 160°F to about 220°F, cooling the extrudate, and comminuting it.

DETAILED DESCRIPTION OF THE INVENTION

55 [0011] For the purpose of describing the proportions of components in the compositions of this invention, the term resin includes the resin *per se* and the curing agent but not the catalyst. Also, the term "one component coating powder" means that the powder is fully formed by grinding and screening only one extrudate of a mixture of the resin, curing agent, catalyst, and additives.

[0012] Epoxy resins which are suitable for the purposes of this invention have an equivalent weight of from about 100 to about 700. Mixtures of such epoxy resins may be used. A suitable mixture may comprise an epoxy resin having

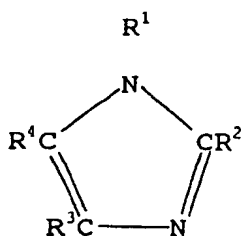
an equivalent weight between about 100 and 400 and on having an equivalent weight between 400 and about 700 in a weight ratio of from 1:99 to 99:1. The resins are exemplified by, but not limited to, those produced by the reaction of epichlorohydrin and a bisphenol, e.g., bisphenol A and bisphenol F. The low melt viscosities of these resins facilitate the extrusion of them in admixture with a curing agent, additives and pigments at about 160-220°F. The preferred melt viscosity is from about 300 to about 1000 centipoise. The melt viscosity of resins having a low T_g , i.e., from about 35°C to about 55°C, is suitable for the purposes of this invention. Epoxy resins known as EPN (epoxy phenol novolac) and ECN (epoxy cresol novolac) resins and those made by the reaction of isopropylidenediphenol (bisphenol A) with epichlorohydrin are suitable for the purposes of this invention. Bisphenol A epoxies are sold under the trademarks ARALDITE GT-7071, GT-7072, EPON 1001 and EPON 2042. ARALDITE GT-6259 is the trademark for an ECN resin.

[0013] The use of a crystalline epoxy may improve the flow characteristics of the fused coating powder. A particularly desirable flow is achieved when a crystalline epoxy constitutes from about 5 to about 20 % by weight of the total amount epoxy resin used in the formulation of the powder. The performance of a coating powder of this invention deteriorates as the level of crystalline epoxy resin therein is increased because of the relatively low equivalent weights of such resins and the preferred amount of such a resin is about 10% or less for that reason. A crystalline epoxy resin having a melting point between about 80°C and about 150°C is preferred. A crystalline epoxy resin having an equivalent weight of about 185 and sold by Shell under the trademark RSS 1407 is suitable for the purposes of this invention.

[0014] When resins having a T_g of from about 35°C to about 40°C, are used in this invention, sintering of the powder is avoided by allowing the temperature in the extruder to rise to activate the low temperature curing agent for a time sufficient to raise the extrudate's viscosity beyond the sintering point and then cooling the extrudate rapidly to about 10-20°C (about 50-70°F) before chipping and grinding it and storing the powder at such temperature to avoid a further viscosity build-up by continued curing. Another way to avoid sintering of the powder when low T_g resins are used is to pre-mix the resin with a crystalline or non-crystalline curing agent powder having an average particle size of about 5 microns that does not liquefy in the extruder. The powdered agent does not react with the resin so readily as does the same curing agent in flake form. A specific example of a curing agent that may so used in the powdered form is sold under the trademark ANCAMINE 2441.

[0015] The low temperature curing agent of this invention is one that will be active at a temperature of from about 225 to 300°F and may be selected from among the many that are commercially available but an epoxy adduct of an aliphatic polyamine (including cycloaliphatic polyamines) having a primary, secondary, or tertiary amino group or a combination of such amino groups is a suitable curing agent for the purposes of this invention. Examples of such curing agents include hardeners sold under the trademarks PF LMB 5218 (Ciba Ceigy), ANCAMINE 2337 XS, ANCAMINE 2014 AS, and ANCAMINE 2441 (Air Products & Chemicals). An epoxy adduct of an aromatic polyamine, such as methylene dianiline, is also a suitable curing agent for the purposes of this invention. It is preferred that the functionality of the adducting reaction mixture is 2 or less and it is particularly preferred to use a difunctional epoxy compound. The amount of low temperature curing agent is from about 2 to about 40 parts per hundred parts of the resin (phr) and the preferred amount is from about 5 to about 20 phr. Increasing levels of the curing agent reduce the gel time and, therefore, increase the orange peel effect.

[0016] A catalyst may be used at a level of from about 0.1 to about 5 parts per hundred parts of the resin, preferably about 0.2 -2 phr to accelerate the curing reaction with the low temperature curing agent. Preferred catalysts for this invention are imidazoles and epoxy adducts thereof, the imidazoles having the general formula:



wherein R^1 , R^2 , R^3 , and R^4 are independently hydrogen, alkyl, aryl, or any substituent which is not reactive with the epoxy resin. For the purposes of this invention, the term imidazole is used herein to mean both the substituted and unsubstituted imidazoles. Imidazoles, in themselves, tend to be insoluble in epoxy resins. Thus, epoxy adducts are made to make them more compatible with the epoxy system of this invention. Suitable adducts of such imidazoles with a bisphenol A epoxy resin are available commercially from Shell Chemical Company under its trademark EPON, e.g., EPON P-101, and also from Ciba-Geigy Corporation under its designation HT 261. Examples of suitable imidazoles include imidazole, 2-methyl imidazole, and 2-phenyl imidazole. For enhanced color stability, the 2-phenyl imidazole,

which is available from the SWK Chemical Co., is preferred. A particularly hard surface is obtained when a combination of the ANCAMINE 2441 curing agent and 2-phenyl imidazole is used to cure an epoxy resin having a medium T_g and melt viscosity.

[0017] Although applicants are not bound by any theory, it is believed that an imidazole adducts to an epoxy resin by an opening of the epoxy ring that results in the epoxy oxygen bonding to the C=N bond of the imidazole ring. The adducted imidazole moves from one epoxy group to another as it facilitates epoxy ring openings and cure reactions.

[0018] A tertiary amine such as triethylamine diamine, available under the trademark ACTIRON SI 27071 from Synthron Chemicals, is another type of curing catalyst that may be used in this invention.

[0019] Various gloss levels for the cured coating may be achieved through the choice of epoxy resins, curing agents, curing catalysts and the relative amounts of each. A low gloss may be achieved, for example, by the use of a combination of dicyandiamide (a slow acting agent) and a fast acting agent to set up competitive reactions. A fused and thermally cured powder coating made from an epoxy resin having an equivalent weight of about 400 may have a 60° gloss level as low as about 10 whereas a gloss level of about 90 may be achieved when the equivalent weight is about 650 and the curing agent and curing catalyst are changed.

[0020] The coating powder may also contain a flow control agent in the range of from about 0.5 to about 2.0 phr. Examples of the flow control agents include the MODAFLOW poly(alkylacrylate) products and the SURFYNOL acetylenic diols; they may be used singly or in combination. Anti-oxidants may also be used at a concentration of from about 0.5 to about 2.0 phr to prevent the discoloration of the coatings even at the relatively low curing temperatures suitable for the purposes of this invention. Examples of the anti-oxidants that are useful in this invention include sodium hypophosphite, tris-(2,4-di-t-butyl phenyl) phosphite (sold under the trademark IRGAFOS 168), and calcium bis[(monoethyl(3,5-di-t-butyl-4-hydroxybenzyl)phosphonate)] (sold under the trademark IRGANOX 1425). Mixtures of anti-oxidants may be used.

[0021] The powder of this invention may be used in coating glass, ceramics, and graphite-filled composites as well as metallic substrates such as steel and aluminum. Much heat is wasted by heating thick or massive metal articles in order to raise the surface temperature to the high levels required by conventional coating powders. The particular utility of the powder of this invention, however, in the coating of heat sensitive substrates such as plastics, paper, cardboard and woods makes it highly appealing as a commercially viable alternative to the liquid coatings that have been almost universally used in the past. For the purposes of this invention, wood is defined as any lignocellulosic material whether it comes from trees or other plants and whether it be in its natural form, milled, or made into plywood, particleboard, or fiberboard of various densities. It is exemplified by lumber, panels, molding, siding, oriented strand board, hardboard, and medium density fiberboard (MDF). The particle board may be standard or treated to enhance its electrical conductivity. Wood having a moisture content of from 3 to 10 % by weight is suitable for the purposes of this invention. A porous particleboard, pre-coated with a conductive liquid coating composition and cured, may also serve as a substrate for the coating powder of this invention. For example, a smooth 2-3 mil thick powder coating is achieved on a 0.5 to 1 mil thick UV or thermally cured pre-coat.

[0022] Pigments, optical brighteners, fillers such as calcium carbonate and bentonite clays, texturizing agents such as particulate rubber, and other conventional additives may also be present. A particularly desirable textured finish may be obtained by the addition of from about 14 to about 20 phr of the rubber to the coating composition along with calcium carbonate at a rubber to carbonate ratio of from about 0.7:1 to about 1.5:1 by weight. Titanium dioxide, in an amount of from about 5 to about 50 phr or more, is an example of a pigment that may be used. An optical brightener, exemplified by 2,2'-(2,5-thiophenediyl)bis[5-t-butylbenzoxazole, sold under the trademark UVITEX OB, may be present at from about 0.1 to about 0.5 phr.

[0023] The coating powder of this invention may be applied by any of the conventional powder coating methods.

[0024] Flat-surfaced and grooved panels may be coated by triboelectric guns on a flat line conveyor having electrically conductive bands around the circumference of the conveyor belt. A suitable flat line powder coating apparatus comprises such a conveyor extending through a powder coating booth, wherein a wooden article supported and moved by the conveyor belt is coated triboelectrically by a plurality of guns situated adjacent one another and in one or more tiers. The article bearing the powder is then conveyed through a curing oven having several heating zones, some of which are heated by IR lamps, others by heat convection, and still others by a combination of those two. The coating and curing line speeds may be the same or different depending on the length of the curing oven. The line speed through the powder application booth may be from about 5 to about 150 feet per minute but it is preferably from about 20 to about 100 feet per minute. The line speed through the curing oven, on the other hand, may be from about 5 to about 20 feet per minute, depending on the oven temperature and the particular coating powder used. The curing temperature may range from about 225° up to but not including the decomposition temperature of the powder. It is preferred to maintain the cure temperature within the range of from about 225° to about 300°F and still more preferred to keep the cure temperature at from about 225 to about 250°F. It is preferred that the coating and curing line speeds be adjusted to the oven length so that they are balanced.

[0025] Preheating of the panel before the coating step is preferred in some instances, e.g., to help the powder reach

its flow temperature in the first zone of the oven and it also minimizes outgassing during cure. The oven may have several heating zones of the IR and convection types and also a combination of the two.

[0026] The film thickness of the cured coating is at least about 1 mil and it may be as much as about 8 mils or even higher if there is a practical need for such. Film thicknesses of from about 4 to about 6 mils are achieved regularly by the method of this invention.

[0027] The gel time of the coating powder of this invention was measured according to ASTM Specification D-3451 (14) in which a small quantity of powder was dropped onto a hot plate at 300°F (149°C) and stroked with a tongue depressor until continuous and readily breakable filaments were formed when the depressor was lifted from the sample. The elapsed time for this to occur was measured in seconds and is the gel time.

[0028] The blocking resistance of a coating powder is tested by placing about 1 inch of the powder in a tube, placing a 100 gram load on top of the powder and heating it at 110°F for 24 hours. Upon removal of the contents of the tube, the degree of sintering is measured on a scale of 1 to 10, 1 being completely free-flowing and 10 being non-flowing. Powders having a value of less than 5 are acceptable.

[0029] The hot plate melt flow (HPMF) of the powder coating composition of this invention was measured by placing a pellet of powder having a diameter of 12.7mm and 6mm thick on a hot plate set at 375°F (190 ± 2°C) at an inclination angle of 35°. When the pellet melts and runs down the plate, the length of the flow is measured in millimeters. The flow distance is dependent on the initial melt viscosity, the rate of reaction, the temperature at which the test is conducted, and the type and amount of catalyst.

[0030] The Hoffman scratch resistance of the coated articles made according to this invention was measured with the Byk-Gardner scratch tester.

[0031] The Taber abrasion resistance of the coated articles made according to this invention was measured according to ASTM D-4060 using CS-10 wheels and a 1000 gram load.

[0032] The invention is more specifically described in the following working examples wherein parts are by weight unless otherwise stated.

EXAMPLES 1-3

[0033] Three resins of increasing viscosity and increasing T_g, as shown in Table A, were melt compounded with polyamine adducts and the other components shown in Table 1 in a twin screw extruder. The extrudate was cooled between water cooled rollers, broken into chips, and then ground into a powder. Powder passing through a 200 mesh screen was electrostatically coated on cold rolled steel Q-panels and cured at 300°F for 5 minutes to obtain a film thickness of 2-3 mils. As shown in Table 2, the superior solvent resistance and blocking resistance of Examples 1-3 demonstrates that faster cure is achieved using this technology. The extrudate temperatures and the properties of the uncured and cured powders are given in Table 2.

TABLE A

Curing agent	T _g °C	Viscosity (cps at 150°C)
GT-7071	38	500
GT-7072	54	1100
GT-7013	60	2700

TABLE 1

Component	Example 1	Example 2	Example 3
GT-7071	85		
GT-7072		85	
GT-7013			85

EP 0 916 709 A2

TABLE 1 (continued)

Component	Example 1	Example 2	Example 3
Curing Agent *	15	15	15
Catalyst **	2.0	2.0	2.0
Titanium Dioxide	30.0	30.0	30.0
Antioxidant ***	1.0	1.0	1.0
SURFYNOL 104S	1.0	1.0	1.0
MODAFLOW 2000	1.0	1.0	1.0
UVITEX OB	0.1	0.1	0.1

* LMB 5218 Epoxy/Polyamine;

** EPON P-101 Epoxy/Imidazole;

*** Sodium Hypophosphite

TABLE 2

Properties	Example 1	Example 2	Example 3
Extrudate temp	160 °F	180 °F	200 °F
Gel time(300°F)	30 sec	23 sec	22 sec
HPMF (375°F)	80 mm	15 mm	13 mm
Blocking	2	2	2
MEK Resistance	4	4	4
60° Gloss	88	56	50
Orange Peel	Slight	Heavy	Heavy

[0034] Table 2 shows the significant reduction in the temperature that the extrudate reaches when a low viscosity epoxy resin is used. The shorter gel times for the powders containing the more viscous resins are caused by the partial curing of the resin in the extruder at the higher temperatures.

EXAMPLES 4-6

[0035] The use of non-adducted imidazoles as a curing catalyst, the practice of the invention without a curing catalyst, and the use of a high temperature curing agent in combination with a low temperature curing agent and a catalyst are illustrated by the compositions and properties thereof shown in Tables 3 and 4.

TABLE 3

Component	Example 4	Example 5	Example 6
GT 7071 resin	85	80	85
LMB 5218	15	20	15
Imidazole	2		
2-Me-imidazole *			0.2
Dicyandiamide			5.0
TiO ₂	30.0	30.0	30.0
Antioxidant **	1.0	1.0	1.0
SURFYNOL 104S	1.0	1.0	1.0

* Me = methyl

** Sodium Hypophosphite

EP 0 916 709 A2

TABLE 3 (continued)

Component	Example 4	Example 5	Example 6
MODAFLOW 2000	1.0	1.0	1.0
UVITEX OB	0.1	0.1	0.1

TABLE 4

Properties	Example 4	Example 5	Example 6
Extrudate temp	150	- - -	160
Gel Time (300°F)	25 sec	25 sec	26 sec
HPMF (375°F)	150 mm	25 mm	38 mm
Blocking Resistance	3	2	2
MEK Resistance	5	3-4	4
60° Gloss	82	45	34
Orange Peel	Slight	Moderate	Slight

[0036] The properties of the coating powder containing imidazole as the catalyst recommend it for use where fast cures are required, as in the coating of reinforcing bars, for example; it has the best cure, as indicated by its MEK resistance, and its flow properties are extraordinarily good.

Example 7-9

[0037] The following examples illustrate the performance of curing agents containing secondary and tertiary amino groups. The coating powder passing through a 200 mesh screen was sprayed from a tribocharging gun onto 1" thick medium density fiberboard (MDF) panels which had been pre-heated in a 350°F oven for 10 minutes to a surface temperature of 240°F. The powder coating was cured by heating the panels at 350°F for 5 minutes to reach a surface temperature of 280°F.

TABLE 5

COMPONENT	Example 7	Example 8	Example 9
EPON 2042 resin	70		
ANCAMINE 2014 AS	20		
RSS 1407 resin	10		
EPON P-101	2.0		
MODAFLOW 2000	1.0	1.0	1.0
SURFYNOL 104S	1.0	1.0	1.0
Antioxidant *	1.0	1.0	1.0
TiO ₂	30	30	30
ANCAMINE 2441		10	10
2-phenylimidazole			2.0
GT 7072 resin		100	100

* Sodium hypophosphite

TABLE 6

PROPERTY	Example 7	Example 8	Example 9
Gel Time (300°F)	30 sec	90 sec	35 sec
HPMF	43 (mm)	90 (mm)	75 (mm)
MEK Resistance	3	4	5
60° Gloss	35	60	95
Hoffman Scratch Resistance *	1,200 grams	1,000 grams	2,000 grams
Taber Abrasion Resistance	36 mg	72 mg	69 mg

Comparative Example 1

[0038] A two-component epoxy powder coating composition described in Example 9 of commonly assigned, co-pending application filed March 4, 1997 as Serial No. 810,745 was prepared for comparison with the composition of this invention. Components A and B of the formulation shown in Table 7 are extruded separately and then ground and classified in the usual manner. Sixty-five parts by weight of Component A and 35 parts of Component B were blended to form the coating powder composition of Comparative Example 1. The powder of Comparative Example 1 was applied to a wooden panel that had been pre-heated to a panel temperature of 220-230°F and was cured at a panel temperature of 270-280°F for about 5 minutes. The properties of the cured coating are shown in Table 8.

TABLE 7

Ingredient	Component A	Component B
GT-7072 (melt visc. 54)	100	---
2-phenyl imidazole	2.0	---
MODAFLOW 2000	1.0	2.0
SURFYNOL 104S	1.0	30.0
LMB 5218 curing agent	---	100
Titanium Dioxide	30.0	30.0
Polyethylene (Grade 6A)	2.0	2.0
Sodium Hypophosphite	1.0	1.0
Optical brightener	0.1	0.1

TABLE 8

Gel time	25 seconds
HPMF	60 mm
MEK resistance	4
60° gloss	15
Hoffman scratch resistance	500 grams
Taber abrasion	120 mg

Examples 10-12

[0039] The following examples illustrate the combined effects of curing agents having different rates and epoxy resins having low and high equivalent weights on the gloss of powder coatings of this invention. The coating powder passing through a 200 mesh screen was sprayed from a tribocharging gun onto 1" thick medium density fiberboard (MDF) panels which had been pre-heated in a 350°F oven for 10 minutes to a surface temperature of 240°F. The powder coating was cured by heating the panels at 350°F for 5 minutes to reach a surface temperature of 280°F.

TABLE 9

COMPONENT	Example 10	Example 11	Example 12
GT-6259 resin *	100	50	50
GT-7072 resin **	---	50	50
ANCAMINE 2014 AS	20	20	10
ANCAMINE 2441	---	---	5
EPON P-101	2.0	2.0	2.0
MODAFLOW 2000	1.0	1.0	1.0
SURFYNOL 104S	1.0	1.0	1.0
Antioxidant ***	1.0	1.0	1.0
TiO ₂	30	30	30

*Eq Wt. ~400;

** Eq Wt ~650

*** Sodium hypophosphite

TABLE 10

PROPERTY	Example 10	Example 11	Example 12
Gel Time (300°F)	11 sec	11 sec	15 sec
HPMF	48 (mm)	39 (mm)	50 (mm)
MEK Resistance	sl rub off	sl rub off	sl rub off
60° Gloss	15	25	70
Hoffman Scratch	300 grams	300 grams	200 grams
Taber Abrasion	82 mg	64 mg	71 mg
Smoothness	sl OP	sl OP	mod OP

Examples 13-17

[0040] Coating powders formulated as shown in Table 11 were prepared by extrusion through a die set in an extruder having a 12 pin rotor operating at 300 rpm and whose front zone is at 120°C and rear zone is cool. The extrudate is cooled chipped and ground to pass through a 200 mesh screen. The resulting powder was sprayed electrostatically from a tribocharging gun onto 1" thick medium density fiberboard (MDF) panels which had been pre-heated in a 350°F oven for 10 minutes to a surface temperature of 240°F. The powder coating was cured by heating the panels at 350°F for 5 minutes to reach a surface temperature of 280°F. The thickness of the coating was 4-7 mils.

TABLE 11

COMPONENT	Ex 13	Ex 14	Ex 15	Ex 16	Ex 17
GT-6259 resin	100		50	50	50
GT-7072 resin	---	100	50	50	50
ANCAMINE 2014 AS	20	20	20	5	20
ANCAMINE 2441	---	---	5	10	---
EPON P-101	2.0	2.0	2.0	2.0	2.0
MODAFLOW 2000	1.0	1.0	1.0	1.0	1.0
SURFYNOL 104S	1.0	1.0	1.0	1.0	1.0
Antioxidant *	1.0	1.0	1.0	1.0	1.0
TiO ₂	30	30	30	30	30

* Sodium hypophosphite

TABLE 12

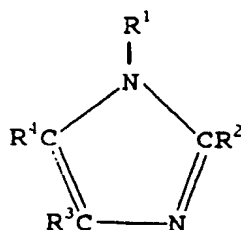
PROPERTY	Ex 13	Ex 14	Ex 15	Ex 16	Ex 17
Extrudate Temp	165-175	180-190	175-180	175-185	175-185
Gel Time (300°F)	11 sec	13 sec	11 sec	15 sec	16 sec
HPMF (mm)	48	36	39	50	88
MEK Resistance	4+	4	4+	5	4+
60° Gloss	15	26	25	70	17
Hoffman Scratch	300 grams	300 grams	300 grams	200 grams	---
Taber Abrasion	82.2 mg	75.9 mg	63.7 mg	71.4	54.3
Smoothness	sl OP	mod OP	sl OP	mod OP	no OP

Claims

1. A method for preparing a thermosetting coating powder which comprises extruding, at a temperature of not more than 104.5°C (220°F), a mixture of an epoxy resin having a melt viscosity of from 0.2 to 2.0 Pas (200 to 2000

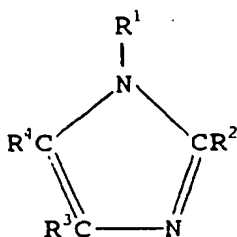
centipoise) and a low temperature curing agent which is solid at 27°C (80°F), cooling the extrudate and comminuting it.

2. A method according to claim 1 wherein the low temperature curing agent is an epoxy resin adduct of a polyamine, preferably an aliphatic polyamine.
3. A method according to claim 1 or claim 2 wherein the curing agent is in powder form.
4. A method according to any preceding claim wherein an imidazole having the general formula:



or an epoxy adduct thereof, wherein R¹, R², R³, and R⁴ are independently hydrogen or any substituent which is not reactive with the epoxy resin, is added to the extrusion mixture as a catalyst.

5. A method according to claim 4 wherein R¹, R², R³, and R⁴ are independently hydrogen, alkyl, aryl, or alkaryl.
6. A method according to claim 5 wherein R² is methyl or phenyl, and R¹, R³, and R⁴ are hydrogen.
7. A method according to any preceding claim wherein from 5 to 20% by weight of the epoxy resin is crystalline.
8. A method according to any preceding claim wherein the epoxy resin is a mixture of one having an equivalent weight between 100 and 400 and one having an equivalent weight between 400 and 700 in a weight ratio of from 1:99 to 99:1.
9. A method according to any preceding claim wherein the epoxy resin has an equivalent weight of from 350 to 700.
10. A method according to claim 9 wherein the epoxy resin has an equivalent weight of about 400.
11. A method according to claim 9 wherein the epoxy resin has an equivalent weight of about 650.
12. A method according to any preceding claim wherein the extrusion temperature is from 71 to 104°C (160 to 220°F).
13. A thermosetting coating powder which cures at a temperature of from 107 to 149°C (225 to 300°F) and comprises an epoxy resin having a melt viscosity of from 0.2 to 2.0 Pas (200 to 2000 centipoise) at 150°C and a low temperature curing agent which is solid at about 27°C (80°F).
14. A powder according to claim 13 wherein the low temperature curing agent is an epoxy resin adduct of an aliphatic polyamine.
15. A powder according to claim 14 wherein the aliphatic polyamine contains a primary amino group.
16. A powder according to claim 14 or claim 15 wherein the aliphatic polyamine contains a secondary amino group.
17. A powder according to any one of claims 13 to 16 wherein the amount of curing agent is from 2 to 40 parts per hundred parts of the resin.
18. A powder according to any one of claims 13 to 17 further characterized in that it contains an imidazole having the general formula:



or an epoxy adduct thereof wherein R¹, R², R³, and R⁴ are independently hydrogen or any substituent which is not reactive with the epoxy resin.

19. A powder according to claim 18 wherein R¹, R², R³, and R⁴ are independently hydrogen, alkyl, aryl or alkaryl.
20. A powder according to claim 19 wherein R² is methyl or phenyl, and R¹, R³, and R⁴ are hydrogen.
21. A powder according to any one of claims 18 to 20 wherein the amount of imidazole or epoxy adduct thereof is from 0.2 to 5 parts per hundred parts of the resin.
22. A powder according to any one of claims 13 to 21 wherein the epoxy resin has an equivalent weight of from 100 to 700.
23. A powder according to claim 22 wherein the epoxy resin has an equivalent weight of about 400.
24. A powder according to claim 22 wherein the epoxy resin has an equivalent weight of about 650.
25. A powder according to any one of claims 13 to 24 wherein the epoxy resin is a mixture of one having an equivalent weight between 100 and 400 and one having an equivalent weight between 400 and 700 in a weight ratio of from 1:99 to 99:1.
26. A powder according to any one of claims 13 to 25 wherein from 5 to 20 by weight of the epoxy resin is crystalline.
27. A method for coating wood comprising electrostatically spraying a thermosetting coating powder according to any one of claims 13 to 25 onto a surface of the wood and heating the powder to a curing temperature of from 107 to 149°C (225 to 300°F).
28. An article having a thermosetting epoxy powder coating composition powder coated and cured thereon, which composition in powder form comprises a particulate blend of: an epoxy resin having a melt viscosity of from 0.2 to 2.0 Pas (200 to 2000 centipoise) at 150°C and a low temperature curing agent which is solid at about 27°C (80°F).
29. An article according to claim 28 wherein the amount of curing agent is from 2 to 40 parts per hundred parts of the resin.
30. An article according claim 28 or claim 29 wherein the low temperature curing agent is an epoxy resin adduct of an aliphatic polyamine.
31. An article according to any one of claims 28 to 30 wherein the epoxy resin has an equivalent weight of from about 100 to 700.
32. An article according claim 31 wherein the epoxy resin has an equivalent weight of about 400.
33. An article according to claim 31 wherein the epoxy resin has an equivalent weight of about 650.
34. An article according to any one of claims 31 to 33 wherein the epoxy resin is a mixture of one having an equivalent weight between 100 and 400 and one having an equivalent weight between 400 and 700 in a weight ratio of from 1:99 to 99:1.

35. An article according to any one of claims 28 to 34 wherein the article is made of wood.

5

10

15

20

25

30

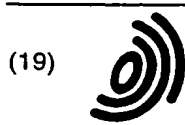
35

40

45

50

55



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) **EP 0 916 709 A3**

(12) **EUROPEAN PATENT APPLICATION**

(88) Date of publication A3:
08.11.2000 Bulletin 2000/45

(51) Int Cl.7: **C09D 163/00, C09D 5/03,
C08G 59/18**

(43) Date of publication A2:
19.05.1999 Bulletin 1999/20

(21) Application number: **98308757.8**

(22) Date of filing: **27.10.1998**

(84) Designated Contracting States:
**AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE**
Designated Extension States:
AL LT LV MK RO SI

(30) Priority: **04.11.1997 US 964242
07.07.1998 US 111419**

(71) Applicant: **MORTON INTERNATIONAL, INC.
Chicago, Illinois 60606 (US)**

(72) Inventors:
• **Muthiah, Jeno
Wernersville, Pennsylvania 19565 (JP)**

• **Teti, Jeremiah J.
Reading, Pennsylvania 19606 (JP)**
• **Schlessman, Jacquelyn M.
Temple, Pennsylvania 19560 (JP)**

(74) Representative:
**Bankes, Stephen Charles Digby et al
BARON & WARREN
18 South End
Kensington
London W8 5BU (GB)**

(54) **A one-component, low temperature curable coating powder**

(57) A mixture of a self-curing epoxy resin having an equivalent weight of from 100 to 700, a melt viscosity of from 200 to 2000 centipoise at 150°C and a low temperature curing agent is extruded as one component at a

temperature below 105°C and the extrudate is cooled and pulverized to form a low temperature curable coating powder. The powder cures at a temperature of from 107 to 149°C and produces a coating having an exceptionally smooth surface with either a low or high gloss.

EP 0 916 709 A3



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 98 30 8757

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	US 5 414 058 A (ONO ET AL) 9 May 1995 (1995-05-09) * column 1, line 53 - column 5, line 24; claim 1; example 1 *	1,4,7	C09D163/00 C09D5/03 C08G59/18
X	GB 1 583 539 A (DU PONT DE NEMOURS) 28 January 1981 (1981-01-28) * page 2, line 9 - page 5, line 11; claim 1; example 1 *	1	
P,X	EP 0 806 445 A (MORTON INTERNATIONAL) 12 November 1997 (1997-11-12) * page 2, line 53 - page 3, line 57; claims 1-23; examples *	1-35	
A	GB 2 118 188 A (VALENTINE VARNISH AND LACQUER COMPANY) 26 October 1983 (1983-10-26) * page 1, line 28 - line 86 *	1,13	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
			C08G C09D
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 18 September 2000	Examiner Bourgonje, A
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

EPO FORM 1503 03/02 (P4/C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 98 30 8757

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

18-09-2000

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5414058 A	09-05-1995	CA 2043288 A	29-11-1991
		JP 2544686 B	16-10-1996
		JP 4288317 A	13-10-1992
		KR 159525 B	15-12-1998
GB 1583539 A	28-01-1981	CA 1103387 A	16-06-1981
		DE 2826556 A	04-01-1979
		ES 470830 A	16-09-1979
		FR 2394590 A	12-01-1979
		JP 54007437 A	20-01-1979
		NL 7806481 A	19-12-1978
		US 4122060 A	24-10-1978
EP 806445 A	12-11-1997	US 5714206 A	03-02-1998
		CA 2202336 A	06-11-1997
		EP 0942050 A	15-09-1999
		US RE36742 E	20-06-2000
		US 5907020 A	25-05-1999
		US 6077610 A	20-06-2000
GB 2118188 A	26-10-1983	NONE	

EPO FORM P4489

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82